



Title: Quality Assurance and Quality Control Requirements and Performance Standards
for SW-846 7000 Series Atomic Absorption Methods for Metals

WSC – CAM – III C

Quality Assurance and Quality Control Requirements and Performance Standards for **SW-846 7000 Series**, Atomic Absorption Methods for Metals for the Massachusetts Contingency Plan (MCP)

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III Trace Metal Analyses

C. Quality Assurance/Quality Control (QA/QC) Requirements and Performance Standards for SW-846 7000 Series, Atomic Absorption Methods for Metals

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1.0 QA/QC Requirements for SW-846 Series 7000 Methods

1.1 Overview of Method

Atomic absorption (AAS) spectroscopy determines metals in solution. Drinking water free of particulate matter may be analyzed directly; however, groundwater, other aqueous samples, TCLP extracts, industrial wastes, soils, sludges, sediments, and other solid wastes require digestion prior to analysis. Solubilization and digestion procedures are presented in Chapter 3, Section 3.2 of SW-846 and summarized herein in Appendix III C-2. Analysis for dissolved metals does not require digestion if the sample has been filtered and acidified; however, digestion may be beneficial to reduce potential matrix effects even in dissolved (filtered) samples.

In most cases, direct-aspiration (or flame) atomic absorption techniques do not provide adequate sensitivity for purposes of MCP decision making. In these cases, Inductively Coupled Plasma- Atomic Emission Spectrometry (ICP-AES) or the graphite furnace AAS is utilized. In some cases (e.g., for seawater or brackish water samples) other specialized analytical procedures such as the gaseous-hydride AAS method for arsenic and selenium may be required to enhance sensitivity or to overcome interferences. In current practice, ICP-AES is the method of choice for most trace metal determinations (excluding mercury). For MCP work, Graphite Furnace AAS (GFAA) is generally limited to the analysis of water samples for arsenic, lead, thallium and selenium because it can be more sensitive than some ICP-AES instruments for these analytes in this matrix.

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for metals analyzed in support of MCP decision-making are presented in Appendix III C-1 of this document and Appendix VII-A, CAM-VII A, "Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)".

1.1.1 Direct Aspiration

In Flame AAS (FLAA) direct-aspiration determinations, a sample is aspirated and atomized in a flame. A light beam from a hollow cathode lamp or an electrodeless discharge lamp is directed through the flame into a monochromator, and onto a detector that measures the amount of absorbed light. Absorption depends upon the presence of free unexcited ground-state atoms in the flame. Because the wavelength of the light beam is characteristic of only the metal being determined, the light energy absorbed by the flame is a measure of the concentration of that metal in the sample. This principle is the basis of atomic absorption spectroscopy. Direct-aspiration atomic absorption spectroscopy determinations, as opposed to ICP, are normally completed as single element analyses and are relatively free of inter-element spectral interferences. Either a nitrous-oxide/acetylene or air/acetylene flame is used as an energy source for



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dissociating the aspirated sample into the free atomic state, making analyte atoms available for absorption of light. In the analysis of some elements, the temperature or type of flame used is critical. If the proper flame and analytical conditions are not used, chemical and ionization interferences can occur.

1.1.2 Furnace Procedure

Graphite Furnace AAS (GFAA) replaces the conventional flame with an electrically heated graphite furnace. The furnace allows for gradual heating of the sample aliquot in several stages. Thus, the processes of dissolution, drying, decomposition of organic and inorganic molecules and salts, and formation of atoms which must occur in a flame or ICP in a few milliseconds may be allowed to occur over a much longer time period and at controlled temperatures in the furnace. The measurement principle for this technique is essentially the same as with direct aspiration atomic absorption, except that a furnace, rather than a flame, is used to atomize the sample. As a greater percentage of available analyte atoms is vaporized and dissociated (atomized) in the tube as compared to a flame, the use of smaller sample volumes and detection of lower concentrations of elements is possible with the furnace technique. Because of this increased sensitivity, interferences sometimes can present analytical difficulties. By optimizing digestion, heating times and temperatures, and matrix modifier combinations, an experienced analyst can remove unwanted matrix components and minimize interferences.

1.1.3 SW-846 Methods 7000A Interferences

Samples submitted to a laboratory for trace metal analysis may become contaminated by numerous routes during both sampling and analysis. Potential sources of contamination from sampling and processing may include:

- Metallic or metal-containing containers and sampling equipment,
- Laboratory acids or reagents,
- Improperly cleaned or stored equipment, and
- Atmospheric inputs such as dirt and dust

Physical interferences, chemical interferences, and ionization interferences can affect the accuracy of results for both direct aspiration and graphite furnace AA. Recommended corrective measures, for example, the use of matrix modifiers and graphite platforms to minimize matrix effects, are detailed in Section 3.0 of SW-846 7000 Series Methods.

1.2 General QA/QC Requirements for SW-846 7000 Series Methods

Each laboratory that uses SW-846 7000 Series Methods is required to operate a formal quality assurance program to demonstrate the precision and bias of the method as performed by the laboratory and procedures for determining the method reporting limit (RL). The minimum requirements of this program consist of an initial demonstration of



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laboratory proficiency, ongoing analysis of standards and blanks as a test of continued performance, and the analysis of laboratory control spikes (LCSs) and matrix spikes (MS), to assess accuracy and/or precision. Matrix duplicates or matrix spike duplicates (MSD) may also be used to evaluate precision when such samples are analyzed either at discretion of laboratory or at request of data-user.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Section 1.4 and Table III C-1 of this method. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Chapter One, Section 4.4.1 and SW-846 7000 Series Methods, Section 8.0. The data associated with the Initial Demonstration of Proficiency must be kept on file at the laboratory and made available to potential data-users on request. The data associated with the Initial Demonstration of Proficiency for SW-846 7000 Series Methods must include the following:

QC Element	Performance Criteria
Initial Calibration	CAM-III C, Table III C-1
Continuing Calibration	CAM-III C, Table III C-1
Method Blanks	CAM-III C, Table III C-1
Instrument QC Samples	CAM-III C, Table III C-1
Percent Recovery for MS/LCS	CAM-III C, Table III C-1
Percent Relative Standard Deviation for Duplicates	CAM-III C, Table III C-1

It is essential that laboratory-specific performance criteria for LCS and the other data quality indicators, listed in Table III C-1, also be calculated and documented. When experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices, the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent quality control performance standards described in Table III C-1 to assess analytical trends (i.e., systematic bias, etc) and to improve overall method performance.

For the SW-846 7000 Series Methods, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed on Table III C-1. It should be noted that the performance standards listed in Table III C-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory. Laboratories are encouraged to continually strive to minimize variability and improve the accuracy and precision of their analytical results. The LSP will utilize this analytical performance data to verify that the results reported by the laboratory are consistent with the pre-established data quality objectives for the disposal site.



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Use of this method is restricted to use by, or under the supervision of, analysts who are knowledgeable in atomic absorption spectrophotometry and the correction of interferences described in Section 3, SW-846 Method 7000.

1.2.1 General Quality Control Requirements for Determinative Inorganic Methods

Refer to SW-846 Chapter One for general quality control procedures for all inorganic methods, including SW-846 7000 Series Methods. These requirements ensure that each laboratory maintain a formal quality assurance program and records to document the quality of all inorganic data.

Quality Control procedures necessary to evaluate the instrument's operation may be found in Chapter One, Section 2.0, and include evaluation of calibrations and performance of sample analyses.

Instrument quality control and method performance requirements for atomic absorption analytical systems may be found in SW-846 Method 7000 series.

1.2.2 Specific QA/QC Requirements and Performance Standards for SW-846 7000 Series Methods

Specific QA/QC requirements and performance standards for SW-846 7000 Series Methods are presented in Table III C-1. Strict compliance with the QA/QC requirements and performance standards for these methods, as well as satisfying other analytical and reporting requirements will provide an LSP with "Presumptive Certainty" regarding the usability of analytical data to support MCP decisions. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty", parties must:

- Comply with the procedures described and referenced in CAM-III C;
- Comply with the applicable QC requirements prescribed in Table III C-1 for this test procedure;
- Collect and analyze field QC samples at the frequencies prescribed in the CAM-VII A and Appendix III C-2 for this test procedure; and
- Adopt the reporting formats and elements specified in the CAM-VII A.

In achieving the status of "Presumptive Certainty", parties will be assured that analytical data sets:



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- ✓ Will satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data;
- ✓ May be used in a data usability assessment, and if in compliance with all MCP Analytical Method standards, laboratory QC requirements, and field QC recommended limits and action levels, the data set will be considered useable data to support site characterization decisions made pursuant to the MCP; and
- ✓ May be used to help support a data representativeness assessment.

Widespread adherence to the “Presumptive Certainty” approach will promote inter-laboratory consistency and provide the regulated community with a greater degree of certainty regarding the quality of data used for MCP decision-making. The issuance of these requirements and standards is in no way intended to preempt the exercise of professional judgement by the LSP in the selection of alternative analytical methods. However, parties who elect not to utilize the “Presumptive Certainty” option have an obligation, pursuant to 310 CMR 40.0017 and 40.0191(2)(c), to demonstrate and document an overall level of (laboratory and field) QA/QC, data usability, and data representativeness that is adequate for and consistent with the intended use of the data.

Title: **Table III C-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 7000**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Preparation of Samples	Accuracy and Representativeness	All aqueous and solid samples must be prepared prior to analysis, with the exception of aqueous samples filtered for dissolved metals analysis. See SW-846 and Appendix III C-2 for appropriate digestion methods.	No		
Initial Calibration	Laboratory Analytical Accuracy	(1) Frequency – Daily or each time instrument is set up, prior to sample analysis (2) Minimum of a calibration blank plus three calibration standards (3) Low-level standard in calibration must be at the laboratory reporting limit (RL) (4) Linear curve fit with correlation coefficient $r \geq 0.995$. Second order curve fit may be used if $r \geq 0.995$.	No	Recalibrate as required by method.	
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	(1) Frequency - Immediately after each initial calibration (2) Separate-source from calibration standards (3) ICV percent recovery must be 90-110%	No	Recalibrate and/or reanalyze ICV as required by method.	Suspend all analyses until Initial Calibration non-conformance is rectified.
Initial Calibration Blank (ICB)	Laboratory Analytical Sensitivity (instrument drift and contamination evaluation)	(1) Frequency - Immediately after each ICV (2) Must be matrix-matched (the same concentration of acids as standards and samples) (3) ICB must be < Reporting Limit (RL)	No	Recalibrate and/or reanalyze ICB as required by method.	



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Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	(1) Frequency - Every 10 samples and at end of run (2) Same-source as calibration standards; near mid-point of calibration range (3) CCV percent recovery must be 90-110%	No	Recalibrate and/or reanalyze all samples since last compliant CCV.	Narrate noncompliance.
Continuing Calibration Blank (CCB)	Laboratory Analytical Sensitivity (instrument drift and contamination evaluation)	(1) Frequency - Every 10 samples and at end of run immediately after CCV (2) Must be matrix-matched (the same concentration of acids as standards and samples) (3) CCB must be < RL	No	Recalibrate and/or reanalyze all samples since last compliant CCB.	Narrate noncompliance.
Method (Preparation) Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Frequency - One per digestion batch of < 20 field samples. (2) Must be matrix-matched (the same concentration of acids as calibration and QC standards) and digested with the samples (3) Method Blank must be < RL	Yes	Redigest and reanalyze all associated samples unless all detected results are > 10x method blank level.	Narrate noncompliance.
Laboratory Control Sample (LCS)	Laboratory Method Accuracy	(1) Frequency - One per digestion batch of < 20 field samples. (2) LCS must be media-matched (aqueous or solid) to field samples, a separate-source from the calibration standards, and digested with the samples. (3) LCS percent recovery for must be 80-120% for aqueous media and vendor control limits (95% confidence limits) for solids.	Yes	Redigest/reanalyze all associated samples.	Narrate noncompliance. Note: MADEP modification to frequency of LCS for consistency with other methods.

Title: **Table III C-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 7000**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Matrix Spike Sample (MS)	Method Accuracy in Sample Matrix	(1) Frequency - One per digestion batch of < 20 field samples. (2) Percent recoveries must between 75 -125 for all media. (3) Laboratories are expected to develop their own in-house control limits for each media, which should fall within the limits listed above.	Yes	No corrective action required.	Narrate noncompliance
Matrix Spike Duplicate Sample*(MSD)	Method Precision in Sample Matrix	(1) Frequency - One per digestion batch of < 20 field samples. (2) MSD relative percent difference (RPD) criteria: aqueous results: $\pm 20\%$; soil and sediment results: $\pm 35\%$.	Yes	No corrective action required.	Narrate noncompliance
Matrix Duplicate Sample* (MD)	Method Precision in Sample Matrix	(1) Optional (may be done in lieu of MSD)- One per digestion batch of < 20 field samples. (2) MD relative percent difference (RPD) criteria recommended: aqueous results > 5x RL: $\pm 20\%$; aqueous results < 5x RL: difference \leq RL; soil and sediment results > 5x RL: $\pm 35\%$; soil and sediment results < 5x RL: difference $\leq 2x$ RL.	Yes, if MD requested by LSP or run by laboratory as routine QA/QC	No corrective action required,	Narrate noncompliance. Note: acceptance criteria consistent with USEPA Region I data validation guidance

Title: **Table III C-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 7000**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Serial Dilution Analysis	Method Accuracy in Sample Matrix	(1) Frequency – One per batch if analyte is at least 25x reporting limit. (2) Results from 5-fold dilution must meet criterion of $\pm 10\%$ difference. (3) If no sample is $\geq 25x$ the RL, then perform a Post Digestion Spike instead.	No	Recommend PDS (see below) to confirm matrix interference. May perform quantitation by Method of Standard Additions (MSA) to obtain more accurate results and/or narrate matrix effect. Refer to method for MSA procedure.	Narrate serial dilution QC out of control. Poor serial dilution result may be an indication of matrix interferences. Narrate actions taken, if any.
Post-Digestion Spike Analysis (PDS)	Method Accuracy in Sample Matrix	(1) Frequency – perform one PDS only if serial dilution is not in control and using GFAA with Zeeman background correction. If GFAA with other background correction (e.g., deuterium arc) a PDS may be required on every sample due to increase in potential interferences. (2) Spike at 2-5x the original sample concentration or at the project-specific action level if sample result is nondetect. Percent recoveries must be between 85-115%.	No	If post-digestion spike recovers outside of criteria, this is an indication of matrix interference. May perform quantitation by MSA to obtain more accurate results and/or narrate matrix effect.	Narrate post-digestion spike out of control. This QC is an indication of matrix interferences. Narrate actions taken, if any.
Duplicate Injections (graphite furnace only)	Method Precision	(1) Frequency – All check standards, QC samples, and field samples. (2) RPD must be $< 20\%$ for all detected results. Report average of values.	No	Reanalyze until criterion is met for check standards and QC samples. Narrate noncompliance for field samples.	Narrate noncompliance if RPD not met for field samples. This is an indication of potential matrix interference on precision of result.

Title: **Table III C-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 7000**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting	NA	(1) Non detected values must be reported with the sample-specific reporting limit for each metal. (2) The RL must be \leq the applicable regulatory compliance standard for each metal reported (3) The RL must be verified at least daily with a low-level calibration check standard following the calibration curve or supported by the low-level standard in the calibration curve. (4) Results for soils/sediments must be reported on a dry weight basis for comparison to MCP regulatory standards (5) Sample concentrations that exceed the highest calibration standard must be diluted (in the same acid matrix) to fall within the calibration range when reanalyzed	Yes	Not applicable.	

*It is recommended that a matrix spike duplicate analysis be performed if analytes are not suspected to be detected. A matrix duplicate analysis should be performed if analytes are suspected to be detected.



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2.0 Data Usability Assessment for SW-846 Method 7000 Series Methods

Overall data usability is influenced by uncertainties associated with both sampling and analytical activities. This document provides detailed quality control requirements and performance standards for SW-846 7000 Series Methods which may be used to assess the analytical component of data usability. The sampling component of data usability, an independent assessment of the effectiveness of sampling activities to meet data quality objectives, is not substantively addressed in this document.

3.0 Reporting Requirements for SW-846 7000 Series Methods

3.1 General Reporting Requirements for SW-846 7000 Series Methods

General reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in CAM-VIIA. This guidance document provides recommendations for field QC, as well as the required content of the Environmental Laboratory Report, including

- Laboratory identification information presented in CAM-VII A, Section 2.4.1,
- Analytical results and supporting information in CAM-VII A, Section 2.4.2,
- Sample- and batch-specific QC information in CAM-VII A, Section 2.4.3,
- Laboratory Report Certification Statement in CAM-VII A, Section 2.4.4,
- Copy of the Analytical Report Certification Form in CAM-VII A, Exhibit VII A-1,
- Environmental Laboratory Case Narrative contents in CAM-VII A, Section 2.4.5,
- Chain of Custody Form requirements in CAM-VII A, Section 2.4.6

3.2 Specific Reporting Requirements for SW-846 7000 Series Methods

Specific QA/QC Requirements and Performance Standards for SW-846 7000 Series Methods are presented in Table III C-1. Specific reporting requirements for SW-846 7000 Series Methods are summarized below in Table III A-3 as "Required Analytical Deliverables (**YES**)". These routine reporting requirements should always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as "Required Analytical Deliverables (**NO**)", these data are to be available for review during an audit and may also be requested on a client-specific basis.



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**Table III C-2 Routine Analytical Reporting Requirements for SW-846
7000 Series Methods**

Parameter	Required Analytical Deliverable
Initial Calibration	NO
Initial Calibration Verification (ICV)	NO
Initial Calibration Blank (ICB)	NO
Continuing Calibration Verification (CCV)	NO
Continuing Calibration Blank (CCB)	NO
Method (Preparation) Blank	YES
Laboratory Control Sample (LCS)	YES
Field Matrix Spike Sample (MS)	YES, <u>only</u> if requested by the LSP
Field Matrix Duplicate (MD)	YES, <u>only</u> if requested by the LSP
Field Matrix Spike Duplicate (MSD)	YES, <u>only</u> if requested by the LSP
General Reporting	YES¹
1. Non detected values must be reported with the sample-specific reporting limit.	



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4.0 Regulatory Limits for Metals under 310 CMR 40.000

The most stringent (lowest) MCP Reportable Concentrations (RCs) and Method 1 Standards for metals analyzable by SW- 846 method 7000 are as follows:

Metal	RQ Pounds	RC GW-1 mg/L - (ppm)	RC S-1 mg/kg - (ppm)	Method 1 Groundwater GW-1 unless noted <i>ug/L - ppb</i>	Method 1 Soils S-1/GW-1 <i>ug/g - ppm</i>
Antimony	50	0.006	10	6	10
Arsenic	1	0.05	30	50	30
Barium	100	2	1000	2000	1000
Beryllium	5	0.004	0.7	4	0.7
Cadmium	5	0.005	30	5	30
Chromium (III)	100	0.1	1000	100	1000
Chromium (VI)	100	0.1	1000	50	200
Copper	100	10	1000	NS	NS
Lead	5	0.02	300	15	300
Lithium	10	1	100	NS	NS
Mercury ¹	1	0.001	20	2	20
Nickel	10	0.08	300	80 (GW-3)	300
Phosphorous	1	NA	NA	NS	NS
Potassium	10	NA	NA	NS	NS
Selenium	10	0.05	400	50	400
Silver	50	0.007	100	7 (GW-3)	100
Sodium	5	NA	NA	NS	NS
Thallium	50	0.002	8	2	8
Vanadium	50	0.05	400	50	400
Zinc	50	0.9	2500	900 (GW-3)	2500

NA – Not Applicable

RQ – Reportable Quantity

RC – Reportable Concentration for Groundwater (GW-1) and Soils (S-1)

Method 1 Groundwater – GW-1 Category unless otherwise noted

Method 1 Soils – Category S-1/GW-1 in all cases

NS – No MCP Method 1 Standard has been promulgated by the Department.

¹ Mercury values presented for completeness only. Analyze Mercury by
SW-846 Methods 7470A and 7471A.



Title: Sample Preservation, Container and Analytical Holding Time Specifications for Surface Water, Groundwater, Soil and Sediment Samples Analyzed for Metals by SW-846 7000 Series

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for metal analyses conducted in support of MCP decision-making are summarized below and presented in Appendix VII-A of CAM-VII A, Quality Assurance and Quality Control Guidelines for Sampling, Data Evaluation, and Reporting Activities for the Massachusetts Contingency Plan (MCP). Additional guidance may be found in SW-846, Chapter Three.

Matrix	Sample Container(s) ¹	Preservative	Holding Time ²
Total Metals Groundwater and Surface Water	(1) 1-L Polyethylene Bottle for Total Metals	HNO ₃ to pH < 2,	180 days: all AAS metals
Dissolved Metals Groundwater and Surface Water	(1) 1-L Polyethylene Bottle for field-filtered sample for Dissolved Metals	Filter (0.45 µm) on site; or at the laboratory (prior to acid preservation) within 24 hours of collection HNO ₃ to pH <2,	180 days: all AAS metals
Suspended Metals Groundwater and Surface Water	Submit Suspended Solids on Filter to Laboratory	Filter on site Filter 100 – 500 ml of unpreserved sample	180 days: all AAS metals
Soils and Sediments	(1) 4-ounce glass jar	Cool, 4°C	180 days: all AAS metals
Concentrated Waste Samples	125 mL wide mouth glass or plastic	Cool to 4°C	180 days: all metals except mercury 28 days: mercury

¹ The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised.

² From date of sample collection.



**Title: MINIMUM FIELD QC SAMPLE FREQUENCY IN SUPPORT OF "PRESUMPTIVE CERTAINTY" STATUS
for Aqueous, Soil and Sediment Samples Analyzed for Metals by SW-846 7000 Series**

Parties seeking Presumptive Certainty for metal analyses are required to provide the laboratory with the appropriate volumes and types of quality control (QC) samples as summarized in Table VII A-1. Except for samples that require prior laboratory knowledge (i.e., Matrix Spikes, Matrix Spike Duplicates, drinking water samples, trip blanks/equipment blanks), field QC samples must be submitted to the laboratory as blind samples, using the containers and preservatives specified in CAM-VII A, Appendix VII A-1, and Appendix II A-1 of this document.

MINIMUM FIELD QC SAMPLE FREQUENCY IN SUPPORT OF "PRESUMPTIVE CERTAINTY" STATUS

ANALYTES	METHOD (s)	QC ELEMENT	MEDIA		
			SOIL/SEDIMENT	AQUEOUS	DRINKING WATER
Metals	SW-846 7000 Series Methods	Matrix Spike (MS)	1 per 20 samples ^d	1 per 20 samples ^d	1 per 20 samples ^d
		Field Duplicate	Not Mandatory ^a	Not Mandatory ^a	If analyte detected ^c
		Trip Blank	Not Mandatory ^a	Not Mandatory ^a	Not Mandatory ^a

^a Matrix Spikes must be selected that represent the most significant exposure points to human health and the environment. Separate MS required for sediment if both soil and sediment are sampled.

^b Samples designated "Not Mandatory" are not needed for Presumptive Certainty acceptance of analytical data. However, on a site and project-specific basis, the use of one or more of these and other QC elements (e.g. equipment rinsate blanks, etc.) may be advisable and/or necessary to demonstrate usability of the data, and/or to determine if the data are biased high due to contamination by sampling equipment/storage conditions.

^c Field Duplicate MUST be analyzed for any drinking water sample collected that mercury is detected in the primary sample above the reporting limit (RL). Contingency duplicate samples MUST be collected, consistent with the requirements of Appendix III C-1, for every trace metal sample, including drinking water samples for this purpose.



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SW-846 Method	Method Description
3005	Method prepares ground water and surface water samples for total recoverable and dissolved metal determinations by FLAA, ICP-AES, or ICP-MS. The unfiltered or filtered sample is heated with dilute HCl and HNO prior to metal determination.
3010	Method prepares waste samples for total recoverable metal determinations by FLAA, ICP-AES, or ICP-MS. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid. The method is applicable to aqueous samples, EP and mobility-procedure extracts.
3015	Method prepares aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total recoverable metal determinations by FLAA, GFAA, ICP-AES, or ICP-MS. Nitric acid is added to the sample in a Teflon digestion vessel and heated in a microwave unit prior to metals determination.
3031	Method prepares waste oils, oil sludges, tars, waxes, paints, paint sludges and other viscous petroleum products for analysis by FLAA, GFAA, and ICP-AES. The samples are vigorously digested with nitric acid, sulfuric acid, hydrochloric acid, and potassium permanganate prior to analysis.
3040	Method prepares oily waste samples for determination of soluble metals by FLAA, GFAA, and ICP-AES methods. The samples are dissolved and diluted in organic solvent prior to analysis. The method is applicable to the organic extract in the oily waste EP procedure and other samples high in oil, grease, or wax content
3050	Method prepares waste samples for total recoverable metals determinations by FLAA and ICP-AES, or GFAA and ICP-MS depending on the options chosen. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either nitric or hydrochloric acid. The method is applicable to soils, sludges, and solid waste samples.
3051	Method prepares sludges, sediments, soils and oils for total recoverable metal determinations by FLAA, GFAA, ICP-AES or ICP-MS. Nitric acid is added to the representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to metals determination.
3052	Method prepares siliceous and organically based matrices including ash, biological tissue, oil, oil contaminated soil, sediment, sludge, and soil for total analysis by FLAA, CVAA, GFAA, ICP-AES, and ICP-MS. Nitric acid and hydrofluoric acid are added to a representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to analysis